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L9: Entry 5 of 17

File: USPT

Feb 4, 1997

DOCUMENT-IDENTIFIER: US 5599399 A

TITLE: Baths and process for the chemical polishing of stainless steel surfaces

Brief Summary Text (2):

Chemical polishing of metal surfaces is a well-known technique (Polissage electrolytique et chimique des metaux [Electrolytic and chemical polishing of metals] - W. J. McG. Tegart - Dunod - 1960 - p. 122 et seq.); it consists in treating the metal surfaces to be polished with oxidising baths. Baths comprising a mixture of hydrochloric, phosphoric and nitric acids in aqueous solution (U.S. Pat. No. 2,662,814) are generally employed for the chemical polishing of austenitic stainless steels. To improve the polishing quality, suitable additives are usually incorporated in these baths, such as surface-active agents, viscosity regulators and brightening agents. Thus, U.S. Pat. No. 3,709,824 describes a composition of a bath for the chemical polishing of stainless steel surfaces, comprising, in aqueous solution, a mixture of hydrochloric acid, nitric acid and phosphoric acid, a viscosity regulator chosen from water-soluble polymers, a surfactant, and sulphosalicylic acid as brightening agent.

Brief Summary Text (6):

Consequently, the invention relates to baths for the chemical polishing of stainless steel surfaces, comprising, in aqueous solution, a mixture of hydrochloric acid, nitric acid and phosphoric acid, an optionally substituted hydroxybenzoic acid, at least one quaternary ammonium salt and an additive chosen from perchloric acid and the water-soluble salts of perchloric acid.

Current US Cross Reference Classification (3):252/79.2Current US Cross Reference Classification (4):252/79.4*all pos
1/97*

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L9: Entry 9 of 17

File: USPT

Jul 16, 1991

DOCUMENT-IDENTIFIER: US 5032203 A
TITLE: Apparatus for polishing

Brief Summary Text (5):

For polishing a compound semiconductor crystal base such as GaAs and InP, a mixture solution of etching Br._{sub.2} (bromine) and CH._{sub.3} OH (methanol) has conventionally been used as a polishing agent, to obtain a high polishing efficiency and to secure complete crystallinity of the processed surface.

Brief Summary Text (19):

(4) a surfactant is added to the polishing agent in (1), (2) or (3);

Brief Summary Text (30):

More particularly, fine particles (0.01 to 5 .mu.m in diameter) of at least one compound which do not dissolve in the solution such as SiO._{sub.2}, Cr._{sub.2} O._{sub.3}, Al._{sub.2} O._{sub.3}, ZrO._{sub.2}, SiC, CeO._{sub.2}, Fe._{sub.2} O._{sub.3} and CaCO._{sub.3} may be mixed to the solution in an amount of not more than 10% in order to improve the mechanochemical effect of the NaBrO._{sub.2} solution. Further, surfactants and/or phosphates of an alkali metal may also be added to have further improved polishing results.

Detailed Description Text (14):

The same procedure as in Example 1 was followed except that a polishing solution to which a surfactant (for example, an anionic surfactant) was added in an amount of 0.01 to 3.0% by volume was used. No coating film nor stain which was occasionally observed in Example 1 was formed. When said surfactant is added in a larger amount, the polishing efficiency is likely to be somewhat lowered, but a processed surface of higher quality can be obtained.

Detailed Description Text (16):

A surfactant was added to the polishing agents employed in Examples 2 and 3 in the same manner as in Example 4, then processed surfaces of higher quality could be obtained with high efficiency.

Detailed Description Text (28):

As has been explained above, an aqueous solution of which the base is prepared by dissolving crystalline powders containing NaBrO._{sub.2} in water is capable of etching those compound semiconductors such as GaAs, GaP and InP. The polishing agents prepared from said aqueous solution above by adding surfactant, fine particles which do not dissolve in said aqueous solution or phosphate of an alkali metal permit high quality surfaces (having strain-free and mirror-like surface) to be obtained with high efficiency. Furthermore, the solution has high stability and almost non-toxic to human body and can be used in the same manner as previous polishing agents. Thus, the polishing agents of the present invention can be used advantageously without any care in the manufacturing level in industry.

Current US Cross Reference Classification (1):438/692

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L9: Entry 13 of 17

File: USPT

Apr 19, 1983

DOCUMENT-IDENTIFIER: US 4380490 A

TITLE: Method of preparing semiconductor surfaces

Brief Summary Text (11):

It has been found that semiconductor surfaces having an abrupt dielectric discontinuity between the bulk and the ambient may be obtained by (1) pretreating, (2) chemomechanically polishing the semiconductor surface with a bromine-methanol mixture, and (3) stripping residual layers. If the surface is exposed to the ambient atmosphere after the chemomechanical polish, rinsing with a bromine-methanol solution reestablishes the passivating layer. In a preferred embodiment, the initial bromine content of the solution used for chemomechanical polishing is less than approximately 1.0 percent, by volume, and the semiconductor is silicon or germanium. The method appears especially well suited for use with the <111> surface of silicon. For this surface, the ellipsometrically determined $\langle \epsilon_{sub.2} \rangle$ value is approximately 48.05. With the <100> and <110> surfaces of silicon, $\langle \epsilon_{sub.2} \rangle$ values of approximately 44.75 and 44.62, respectively, were obtained. Values this high have never been obtained before.

Detailed Description Text (5):

The semiconductor was then placed in a cell where the surface preparation could be completed while the surface quality was evaluated. If the semiconductor surface is exposed to the ambient atmosphere during transfer to the cell, it may be rinsed in a Br._{sub.2} -methanol polishing solution to reestablish the passivating layer. For example, 0.05 volume percent bromine in methanol was used with Si and Ge.

Detailed Description Text (9):

Although Br-methanol is a standard polishing agent for Ga-Group V compounds, higher values for $\langle \epsilon_{sub.2} \rangle$ were obtained with the method of this invention. After pretreating by rough polishing with 0.5 volume percent Br-methanol and removing the natural oxides, best results were obtained with the chemomechanical polish using 0.05 volume percent bromine in methanol, diluted to methanol as described above, and rinsing, if exposed to the ambient atmosphere, with Br-methanol and then, after drying, stripping the residual layers. It is believed that better surfaces are obtained because the stripping after the bromine-methanol chemomechanical polish reduces the amount of surface roughness and removes passivating bromide layers.

Detailed Description Text (10):

The results indicate that Br-methanol chemomechanical polishing may be used to prepare smooth semiconductor surfaces and, when followed by a stripping step, to minimize the amount of interface material remaining on the surface after preparation. These two features are probably related. After the surface of a given substrate has been prepared according to the method outlined, certain sequences of chemical treatments induce characteristic sequential changes in $\langle \epsilon_{sub.2} \rangle$ even if the limiting values of $\langle \epsilon_{sub.2} \rangle$ are not as high as the best values obtained. These characteristic changes indicate a common outer layer chemistry and, consequently, lower values of $\langle \epsilon_{sub.2} \rangle$ must be due to microscopic roughness or to bulk material damaged in the polishing. While damaged bulk material can be removed by chemical etching, the elimination of microscopic roughness is more delicate. It is known that roughness is reduced if one constituent of an etch-polish rapidly forms a passive layer that dissolves slowly in another constituent. It is also clear that mechanical motion could aid the latter process of the substrate and passivating layers were much harder and softer, respectively, than the polishing pad so that the passivating layer would be removed more quickly from high spots by the burnishing action of the pad. Bromine is far more electronegative than any of the elemental constituents of the semiconductors specifically mentioned and should react essentially indiscriminately with the semiconductors analyzed to form passivating bromine films. The data show that

bromine-methanol treatments leave residual overlayers that are not completely removed by the methanol diluent of the Br-methanol etch, but require additional stripping procedures for their elimination.

Current US Original Classification (1):
438/692

Current US Cross Reference Classification (4):
252/79.1

Current US Cross Reference Classification (5):
252/79.3

Current US Cross Reference Classification (6):
252/79.4

Current US Cross Reference Classification (7):
252/79.5

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L9: Entry 14 of 17

File: USPT

Jun 1, 1982

DOCUMENT-IDENTIFIER: US 4332649 A

TITLE: Method of polishing glass ware with sulfuric acid and hydrofluoric acid

Brief Summary Text (15):

It is also possible to add small amounts of complexing organic acids such as oxalic acid, tartaric acid or malonic acid to the polishing bath. Thereby more alkali ions in the polishing bath can be maintained in solution and consequently the precipitation can be effected with smaller quantities of fluorosilicic acid and at greater intervals, respectively. Apart from the above-stated complexing acids, other chelating agents for sodium and potassium are also suitable provided that they are capable of withstanding the strongly acidic medium. Examples of these are 1,2,3-triaminopropane, alanine, glycine, amino-barbituric acid-N,N-diacetic acid, nitrilotriacetic acid, 2-sulfoaniline diacetic acid, ethylene diamine tetraacetic acid, as well as the various condensed phosphoric acids such as pyrophosphoric acid, tetramethaphosphoric acid, trimethaphosphoric acid, tripolyphosphoric acid and further substances acting on the same principle.

Brief Summary Text (17):

The electrolytic or electrodialytic removal of the alkali ions from the polishing bath is also possible in a membrane electrolytic cell, the alkali ions migrating from the polishing bath through a semi-permeable membrane into the catholyte. For such electrolytic or electrodialytic methods, membranes are used made of a material which is selectively permeable only for cations, i.e. in this case for sodium and/or potassium. Cation exchange membranes are generally known in technology for electrolysis and electrodialysis. The exchanger material may be, by way of example, a resin on the basis of phenol formaldehyde with pendent sulfonic acid groups or may also be the known exchanger materials on the basis of a styrene resin with sulfonic acid exchanger groups, corresponding to the commercial products "Amberlite" and "Dowex", the latter probably being the best known ones. Acrylic acid resins with the carboxylic acid group as exchanger group and vinyl resins with sulfonic acid exchanger group are also suitable. Finally, the newly developed perfluorated homopolymers and copolymers of the ethylene with pendent sulfonic and carboxylic acid groups and similarly effective substances are also suitable. As the catholyte, a sulfuric acid solution is by way of example suitable, from which the alkali sulfate is then crystallized out and the acid is recycled for polishing or can be put to some other use.

Brief Summary Text (18):

Finally, it is also possible to remove the alkali ions from the polishing bath by means of ion exchangers. These may be polymer products in the acid of hydrogen form or zeolitic molecular sieves or the like. The ion exchanger materials are in this case kept in a column and the polishing bath to be freed of the alkali ions is passed through this bed of ion exchangers. The ion exchangers capture the alkali ions and the regenerated polishing bath can then be recycled into the polishing vessel. Products as were already enumerated above for the membrane materials are suitable as organic polymer cation exchangers. In addition, inorganic ion exchanging substances such as permutites and zeolites are suitable, these being aluminosilicates of varied composition which are capable of exchanging their cationic parts against sodium or potassium or function in the manner of molecular sieves, i.e. they do not allow the cations to pass through their skeleton-like molecular structure.

Detailed Description Text (17):

Wine glasses were to be polished in a polishing bath containing 65% by weight H.sub.2 SO.sub.4 and 1.5% HF at a temperature of 45.degree. C. The polishing bath was continuously circulated by pumping from the polishing vessel, through the electrolytic cell and back to the polishing vessel. The electrolytic cell contained between the electrode chambers a semi-permeable membrane which only allowed the passage of the

alkali ions. The polishing bath was introduced into the anode chamber and a sulfuric acid solution was used as the catholyte. Due to the electrolysis, the alkali ions migrated from the anode chamber into the cathode chamber and there formed alkali sulfate. When the saturation concentration of the sulfates was reached in the catholyte, the same were cooled and crystallized out. By means of this electrolysis, the alkali concentration in the polishing bath could be kept constant for a long duration. In the case of this method, a polishing time of 15 minutes was required. In the polishing bath having constant density, the conductivity can be used as control unit for feeding hydrofluoric acid into the polishing bath.

Current US Original Classification (1):
216/86

Current US Cross Reference Classification (7):
216/97

Current US Cross Reference Classification (8):
216/99

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L9: Entry 15 of 17

File: USPT

Feb 17, 1981

DOCUMENT-IDENTIFIER: US 4251384 A
TITLE: Aluminum polishing compositions

Abstract Text (1):

Aluminum polishing solutions containing phosphoric, nitric and sulphuric acids provide etched finishes if the proportion of sulphuric acid is increased. The invention inhibits such etching by addition to the bath of an aromatic ring compound in which at least 2 hetero atoms are conjugated with the ring such as benztriazole.

Brief Summary Text (6):

We have now discovered that certain aromatic organic compounds have a beneficial effect in reducing the occurrence of transfer etch in aluminium polishing solutions. The presence of such etch inhibitors therefore permits the proportion of sulphuric acid in an aluminium polishing solution to be substantially increased.

Brief Summary Text (10):

The hetero atom may be part of an amino or imino group, hydroxyl group, the keto group of a quinone, or a heterocyclic ring, such as triazole, thiazole or thiadiazole ring.

Brief Summary Text (11):

Preferably the etch inhibitor has a benzene or benzo ring fused to a heterocyclic ring, e.g. a five membered heterocyclic ring, for example, benztriazole ##STR1## is particularly effective. Substituted benztriazoles in which the benzene nucleus is substituted with for example, hydroxyl, alkoxy, amino, nitro, or alkyl groups are also operative as are halo-substituted benztriazoles. Other triazole compounds which may be used include naphthalene triazole and naphthalene bistriazole.

Brief Summary Text (15):

Effective etch inhibitors are readily identified by the presence of an aromatic ring system (usually, but not essentially, a six carbon ring) which is stable in the highly acidic medium, and at least two hetero atoms conjugated or conjugable with the ring. An aromatic system is essential for stability in the aggressive polishing solution.

Compounds lacking an aromatic ring system, such as thiazole, thiadiazole, dimercaptothiadiazole or triazole are ineffective, probably due to instability in the medium. At least two hetero atoms, preferably nitrogen, oxygen or sulphur, especially nitrogen, stabilised by conjugation with the ring, are necessary, probably to provide chelating power.

Brief Summary Text (16):

Apart from the hetero groups, the aromatic ring may be substituted by various other groups including alkyl, haloalkyl, hydroxyalkyl, aminoalkyl, alkenyl, aralkyl, keto alkyl, carboxyalkyl, alkenyl, aralkyl, polyoxyalkylene, phosphonoalkyl, sulphoalkyl and the like. Provided that the essential aromatic nucleus is present, together with the conjugated hetero atoms, the only other necessary limitation is that the compound should be soluble in the bath. This generally implies some limitation of the size of the molecule. For this and for commercial reasons the etch inhibitors of our invention usually contain a total of from 3 to 25 carbon atoms, preferably 4 to 20, most preferably 5 to 18, typically 6 to 15, e.g. 6 to 10. However, many exceptions to this rule will be obvious to those skilled in the art. For example, polyoxyalkylene compounds or other bath soluble polymers which meet the necessary criteria will be effective at any available molecular weight, but will normally break down to smaller units in the bath. Normally alkyl or alkoxy substituents will have from 1 to 4 carbon atoms, e.g. methyl, methoxy, ethyl, ethoxy, butyl or butoxy groups. However bath soluble compounds having alkyl or alkoxy substituents containing up to 20 carbon atoms, or higher, will be operative.

Brief Summary Text (23):

In addition to the foregoing components, polishing baths conventionally contain wetting agents, and these are also preferably present in our novel bath. Any of the wetting agents used hitherto in polishing baths may be employed for example, non-ionic surfactants, such as alkyl polyethers. The wetting agent is normally present in trace quantities of for example up to 0.01% although higher proportions may be used.

Detailed Description Paragraph Table (2):

IN COMPOUND FORMULA CONCENTRATION TRANSFER ETCH	% REDUCTION
	1, 2, 3,
BENZTRIAZOLE ##STR7## 5 gpl 100% BENZOFUROXAN ##STR8## 2 gpl 100% 2, 1, 3,	
BENZOTHIADIAZOLE ##STR9## 2 gpl 100% O-PHENYLENEDIAMINE ##STR10## 1 gpl 90%	
M-PHENYLENEDIAMINE ##STR11## 1 gpl 90% CATECHOL ##STR12## 1 gpl 90% O-AMINOPHENOL	
##STR13## 1 gpl 90% 2-MERCAPTOBENZTHIAZOLE ##STR14## 1 gpl 90% 2-MERCAPTOBENZTHIAZOLE	
##STR15## 1 gpl 80% 2-MERCAPTOBENZOXAZOLE ##STR16## 1 gpl 70% MELAMINE ##STR17## 4 gpl	
60% COMPARATIVE EXAMPLES. 1, 2, 4, TRIAZOLE ##STR18## 10 gpl 0% 2, 5-DIMERCAPTO- 1, 3,	
4-THIADIAZOLE ##STR19## 10 gpl 0% 1, 5- PENTAMETHYLENE TETRAZOLE ##STR20## 10 gpl 0%	

Current US Original Classification (1):252/79.4Current US Cross Reference Classification (3):106/3Current US Cross Reference Classification (5):216/103Current US Cross Reference Classification (6):216/87

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L9: Entry 16 of 17

File: USPT

Oct 25, 1977

DOCUMENT-IDENTIFIER: US 4055458 A

TITLE: Etching glass with HF and fluorine-containing surfactantBrief Summary Text (1):

This invention relates to an improved process for etching, more especially acid polishing and mat etching, glass articles in the presence of fluorine-containing surfactants, the liquid required for this purpose, which contains hydrogen fluoride and which may additionally contain sulphuric acid and/or inorganic fluorides, as NH₄F. HF having added to it a water-soluble and acid soluble wetting agent stable therein, more especially a fluorine-containing surfactant.

Brief Summary Text (2):

The acid polishing of glass, especially lead crystal glass, is normally carried out with heated, aqueous solutions of hydrofluoric acid and sulfuric acid in wide-mesh, acid-resistant baskets of plastic materials or copper. The glass surfaces which have become opaque as a result of grinding are made transparent again and brought to a high luster by virtue of the fact that the glass itself, which contains silicon dioxide, is attacked at its surface and the silicon component is converted into volatile or soluble SiF₄, or H₂SiF₆. The acid polishing process is governed by a number of parameters, for example the composition of the bath, the dip time, the dipping technique and the temperature, these factors having to be adapted to the individual types of glass, for example pressed glass, ground glass, colored glasses, etc. The dipping process is generally repeated several times, and the glass articles are freed from adhering solids, for example calcium and lead sulfates or fluorides, in a subsequent water bath or sulfuric acid bath. Finally, the glass articles are rinsed in a water bath (cf. L. Springer, Sprechsaal fur Keramik-Glas-Email, 87 (1954), page 244).

Brief Summary Text (18):

Fluorine-containing surfactants are particularly suitable for use in the process according to the invention because, by virtue of their chemical inertness, they are totally stable in the etching baths at the reaction temperatures, and by virtue of their extreme surface-active properties need only be used in minimal quantities, for example in quantities of from about 10 to 500 mg/liter and preferably in quantities of about 50 to 300 mg/liter of etching liquid. The surface tension of the etching liquids may even be reduced to values below 20 dyn/cm, so that even the surfaces of different types of glass may be uniformly wetted.

Brief Summary Text (19):

Fluorine-containing surfactants of the kind in question are known per se and are commercially available. From the chemical point of view, they are characterized by the fact that they contain a perfluorinated linear or branched carbon radical R₂F⁺ with from 6 to 20 carbon atoms and preferably with from 6 to 12 carbon atoms, and also a corresponding functional radical which determines whether the wetting agent is anion-active, cation-active or non-ionic and which influences solubility, for example in water and acids (cf. O. Scherer in Fortschritte der chem. Forschung 14, (1970) 2, 212 and O. Lichtenberger in Chim. et Ind. 104 (1971) 7, 815, also M. Woodfine in Chimie et Ind. 101 (1969) 895). Examples are perfluoroalkane sulfonic acids and carboxylic acids, for example C₈F₁₇SO₃H or C₆F₁₃CH₂CH₂SO₃H and C₇F₁₅COOH, and their salts and other derivatives of these basic components, such as alcohols, amides, phosphoric acid derivatives, polyether compounds and others. Other examples are the telomerization products based on tetrafluorethylene and perfluoropropene, the products in question generally being mixtures of homologous compounds.

Brief Summary Text (28):

The above-mentioned surfactants are generally added in quantities of from about 10 to 1000 mg/liter of etching liquid. In the case of some surfactants, favorable results are even obtained with smaller additions, for example with additions of about 5 to 500 mg.

Brief Summary Text (29):

The effect of adding a soluble wetting agent resistant to hydrochloric acid and sulfuric acid, more especially a fluorine-containing surfactant, to etching and polishing baths for glass is that both the evaporation losses of HF and the etching times and the temperature of the etching bath are reduced. In addition, contact faults are considerably reduced, presumably because the bath liquid drains off better from the baskets by virtue of the significant reduction in surface tension. The number of rejects caused by persistently adhering particles of the etching sludge are also reduced because these particles can be rinsed off more easily. In addition, glasses treated in accordance with the invention also show an improved surface finish (luster, smoothness, feel etc.).

Brief Summary Text (31):

The effect of adding these soluble wetting agents resistant to hydrogen fluoride, especially the fluorine-containing surfactants, to the mat etching baths for glass is that both the evaporation losses of HF and also the etching times and reaction temperatures are reduced. In addition, the surfaces are more uniformly wetted, largely irrespective of the composition of the glass.

Detailed Description Text (3):

Pressed crystal glass is acid-polished in an automatic acid-polishing installation consisting of an acid polishing bath, containing sulfuric acid and hydrofluoric acid (60% of H₂SO₄, 10-15% of HF, balance water), a sulfuric acid bath for rinsing off the etching sludge, and a water-rinsing bath. The working temperature of the acid polishing bath is 50.degree.-60.degree. C. The tanks have a volume of 1000 liters and are provided with an effective extraction system. The pressed glass articles to be polished are tightly packed into baskets, introduced into the acid polishing bath and automatically moved therein. They are then introduced into the sulfuric acid bath to rinse off the etching sludge. Finally, they are rinsed in the water bath. Rejects among the pressed lead crystal glass articles are caused mainly by residues of acid adhering persistently to the surface of the glass where it is in contact with the basket which gives rise to faults in the finish. If 100 mg/l of tetraethyl ammonium perfluorooctane sulfonate, in the form of a 5% aqueous solution, is added to the acid polishing bath (= 2 liters of 5% solution for 1000 liters of polishing acid) under otherwise the same working conditions, the points of contact are less noticeable and a better luster is obtained. Similar results are obtained by using tetramethyl ammonium perfluorooctane sulfonate or free perfluorooctane sulfonic acid as the fluorine-containing surfactant.

Detailed Description Text (10):

Mat etching is carried out in the same way as in Example 1. the only difference being that C₈H₁₇SO₃N(C₂H₅)₄ is added to the 50% HF as a fluorine-containing surfactant in a concentration of about 150 mg/liter. The pretreatment step is made superfluous by the addition of this surfactant, in addition to which the etching operation as a whole can be shortened to 3 minutes. Purification of the gases given off by evaporating hydrogen fluoride is easier because, overall, the evaporation losses are smaller.

Detailed Description Text (12):

Mat etching is carried out in the same way as in Example 1, except that the pretreatment stage is left out and C₈H₁₅COONH₄ added to the 70% HF as a fluorine-containing surfactant in a concentration of 300 mg/liter. The etching process lasts 3.5 minutes and no faults are observed.

Detailed Description Text (16):

The test is carried out in the same way as in Example 1, but without the pretreatment step, using 65% hydrofluoric acid which additionally contains 50 mg of a non-ionic fluorine-containing surfactant of the formula: ##STR6## per liter of etching liquid. The mat etching process lasted 3 minutes and no faults were observed.

Current US Original Classification (1):

216/97

Current US Cross Reference Classification (1):

216/99

Current US Cross Reference Classification (2) :

252/79.3

Current US Cross Reference Classification (3) :

252/79.4

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L6: Entry 28 of 38

File: USPT

Apr 19, 1983

US-PAT-NO: 4380490
DOCUMENT-IDENTIFIER: US 4380490 A

TITLE: Method of preparing semiconductor surfaces

DATE-ISSUED: April 19, 1983

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Aspnes; David E.	Watchung	NJ		
Studna; Ambrose A.	Raritan	NJ		

US-CL-CURRENT: 438/692; 134/28, 134/29, 134/3, 252/79.1, 252/79.3, 252/79.4, 252/79.5

CLAIMS:

What is claimed is:

1. A method of treating a semiconductor surface comprising the steps pretreating said surface, said semiconductor being selected from the group consisting of Si, Ge, Ga-Group V compounds, and In-Group V compounds, chemomechanically polishing said surface with a mixture of a halogen and an organic solvent, said mixture consists essentially of bromine and methanol, said polishing step comprises diluting said bromine-methanol mixture from 0.05 volume percent bromine to pure methanol, and stripping residual layers.
2. A method as recited in claim 1 in which said semiconductor is selected from the group consisting of Si and Ge.
3. A method as recited in claim 2 in which said surface is Si<111> and said stripping step comprises flowing solutions of HF in methanol and NH₄OH in H₂O and HF in methanol over said surface.
4. A method as recited in claim 2 in which said surface is Si<110> and said stripping step comprises flowing a solution of buffered HF in methanol over said surface.
5. A method as recited in claim 2 in which said surface is Si<100> and said stripping step comprises flowing a solution of HF in methanol over said surface.
6. A method as recited in claim 1 in which said semiconductor is selected from the group consisting of Ga-Group V compounds.
7. A method as recited in claim 6 in which said compound is GaAs.
8. A method as recited in claim 7 comprising the further step of stripping residual layers with H₂O.
9. A method as recited in claim 1 in which said semiconductor is selected from the group consisting of In-Group V compounds.

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L6: Entry 29 of 38

File: USPT

Jun 1, 1982

US-PAT-NO: 4332649

DOCUMENT-IDENTIFIER: US 4332649 A

TITLE: Method of polishing glass ware with sulfuric acid and hydrofluoric acid

DATE-ISSUED: June 1, 1982

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Salzle; Erich	8000 Muchen 40			DE

US-CL-CURRENT: 216/86; 204/520, 205/527, 205/558, 205/750, 205/770, 210/660, 216/97,
216/99, 422/50

CLAIMS:

I claim:

1. A method of polishing glass articles comprising contacting said articles with a polishing bath containing sulfuric acid and hydrofluoric acid and subsequently rinsing the finally polished articles in a sulfuric acid washing bath and/or water washing bath, wherein during the polishing operation dissolved alkali metal ions selected from the group consisting of sodium and potassium are removed from the polishing bath.
2. The method according to claim 1, wherein the alkali metal ions are continuously removed from the polishing bath.
3. The method according to claim 1, wherein the alkali metal ions are removed batch-wise from the polishing bath.
4. The method according to claim 1, wherein the alkali metal ions are removed intermittently from the polishing bath.
5. The method according to claim 1, wherein the removal of the alkali metal ions is achieved by adding to the polishing bath an amount sufficient of fluorosilicic acid to effect precipitation in said polishing bath of said sodium and potassium ions as sodium- and potassium-silicofluoride, without substantial excess, and separating the precipitated alkali silicofluorides.
6. The method according to claim 5, wherein an acid selected from the group consisting of phosphoric acid and acetic acid is added to the polishing bath.
7. The method according to claim 5, wherein an organic acid selected from the group consisting of malonic acid, oxalic acid and tartaric acid is added to the polishing bath.
8. The method according to claim 5, wherein an acid is added to the polishing bath selected from the group consisting of phosphoric acid, acetic acid and malonic acid, oxalic acid and tartaric acid.
9. The method according to claim 1, wherein the alkali metal ions are removed by

electrolysis in a mercury cell.

10. The method according to claim 1, wherein the alkali ions are removed in an electrolytic cell whose electrodes are separated by a cation semi-permeable membrane.

11. The method according to claim 1, wherein the alkali metal ions are removed by means of a cation exchanger.

12. The method according to claim 1, wherein the polishing bath has a sulfuric acid concentration of 40 to 75%.

13. The method according to claim 12, wherein the polishing bath has a hydrofluoric acid concentration of 3 to 10%.

14. The method according to claim 13, wherein the polishing bath has a temperature of at least 70.degree. C.

15. The method according to claim 1, wherein the sulfuric acid washing bath has a concentration of more than 67% H₂SO₄.

16. The method according to claim 15, wherein the sulfuric acid washing bath has a concentration of 68 to 72% H₂SO₄.

17. The method according to claim 15, wherein the sulfuric acid washing bath has a temperature of 55.degree. to 65.degree. C.

18. The method according to claim 1, wherein the polishing bath has a density which is kept constant and is controllable by determining the hydrofluoric acid concentration in the polishing bath through conductivity measurements, thereby allowing an automatic control of the polishing bath on the basis of the measured value, by introducing hydrofluoric acid when a minimum concentration is obtained and by stopping the supply of hydrofluoric acid when a maximum concentration is achieved.

19. The method according to claim 18, wherein the conductivity measurement is continuously performed.

20. The method according to claim 18, wherein the conductivity measurement is intermittently performed.

Patent Assignment Abstract of Title

Total Assignments: 1
Application #: 10081979
Filing Dt: 02/22/2002

Patent #: NONE

Issue Dt:
PCT #: NONE

Publication #: NONE

Pub Dt:
Inventors: Rajiv K. Singh, Seung-Mahn Lee

Title: Chemical-mechanical polishing slurry for polishing of copper or silver films

Assignment: 1
Reel/Frame: 012645/0683 **Received:** 03/11/2002

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05/02/2002

Pages: 4

Conveyance: ASSIGNMENT OF ASSIGNORS INTEREST (SEE DOCUMENT FOR DETAILS).

Assignors: SINGH, RAJIV K.
LEE, SEUNG-MAHN
Exec Dt: 02/22/2002
Exec Dt: 02/22/2002

Assignee: UNIVERSITY OF FLORIDA
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